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L1: Entry 1 of 2 File: DWPI Sep 4, 1985

DERWENT-ACC-NO: 1985-258933

DERWENT-WEEK: 198542

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TITLE: Water-hardenable inorganic compsn. for tiles etc. - contains cement, gypsum,

acrylate! polymer, water and water-reducing agent for high strength prod.

PATENT-ASSIGNEE:

ASSIGNEE CODE INOUE H INOUI

PRIORITY-DATA: 1984JP-0028234 (February 16, 1984)

Search Selected Search ALL Clear

PATENT-FAMILY:

PUB-NO PUB-DATE LANGUAGE PAGES MAIN-IPC

<u>T JP 60171260 A</u> September 4, 1985 004

APPLICATION-DATA:

PUB-NO APPL-DATE APPL-NO DESCRIPTOR

JP 60171260A February 16, 1984 1984JP-0028234

INT-CL (IPC): C04B 22/00; C04B 24/26; C04B 28/00

ABSTRACTED-PUB-NO: JP 60171260A

BASIC-ABSTRACT:

The compsn. comprises 10-90 wt. pts. of water-hardenable cement e.g. portland cement, etc., 10-90 wt. pts. of water-hardenable gypsum e.g. alpha- or beta-CaSO4.1/2H2O, anhydrous CaSO4 etc., 17-25 wt. pts. of H2O (including amt. of H2O contained in the acrylic water-dispersible organic polymer), 2-16 pts. wt. (calculated in terms of solids content) of acrylic water-dispersible organic polymer exhibiting water-reducing effect, e.g. methylmethacrylate- 2-ethylhexylacrylate copolymer, styrene-butylacrylate copolymer etc., and 0.5-2.0 pts. wt. of water-reducing agent e.g. Na salt of melamine sulphonate-formaldehyde condensate, etc.

USE/ADVANTAGE - The compsn. is suitable for use in prodn. of tile, block, roofing material, interior finishing material, floor material, ceiling material etc. Prods. exhibit high strength and excellent water resistance, incombustibility, weather-proofingness and vibration absorbability without generation of deformation, cracking, expansion and shrinkage.

CHOSEN-DRAWING: Dwg.0/0

TITLE-TERMS: WATER HARDEN INORGANIC COMPOSITION TILE CONTAIN CEMENT GYPSUM POLYACRYLATE POLYMER WATER WATER REDUCE AGENT HIGH STRENGTH PRODUCT

DERWENT-CLASS: A93 L02

CPI-CODES: A12-R01; L02-C05; L02-D07A;

UNLINKED-DERWENT-REGISTRY-NUMBERS: 1767U

POLYMER-MULTIPUNCH-CODES-AND-KEY-SERIALS:

Key Serials: 0004 0203 0044 0231 0306 3152 0486 0487 0495 3034 0502 3013 0530 0537 0565 1276 1278 1517 1737 1962 2012 2504 3251 2604 2605 2609 2615 2629 2679 3255 2691 2694 2696 2698 3275

Multipunch Codes: 014 034 038 04- 05- 051 055 056 06- 074 076 077 080 081 082 09- 139 14- 145 180 185 189 225 230 231 249 27& 397 436 53& 532 533 535 539 540 541 542 543 546 549 551 552 554 567 57& 58& 59& 613 614 616 618 623 626 681 688

SECONDARY-ACC-NO:

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City of New York, State of New York, County of New York

I, Livia Cheung, hereby certify that the following is, to the best of my knowledge and belief, a true and accurate translation of this document, "Hydraulic Inorganic Composition – S60-171260", from Japanese into English.

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PAUL D. RALSTON otary Public, State of New York No. 01 RA6023867 Qualified in Queens County 3.

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(54) Title of the invention	n HYDRAULIC INORGANIC COMPOSITION		
	(21) Japanese Patent A	pplication S	559-028234
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SPECIFICATION

1. TITLE OF THE INVENTION

Hydraulic inorganic composition

2. SCOPE OF PATENT CLAIMS

A hydraulic inorganic composition that is characterized in that it comprises,

- a) 10 90 parts by weight of a hydraulic cement,
- b) 10 90 parts by weight of a hydraulic gypsum,
- c) 17 25 parts by weight of water (including the moisture contained in the acryl water-dispersing organic polymer of d),
- d) 2 16 parts by weight of an acrylic, water-dispersing organic polymer that has a moisture-reducing effect (solid portion converted), and
- e) 0.5 2.0 parts by weight of a moisture-reducing agent.

 3. DETAILED DESCRIPTION OF THE INVENTION

 The present invention relates to a hydraulic inorganic composition that realizes water resistance and high strength without the occurrence of deformation or cracking and that permits thin, hardened bodies with large dimensions to be formed easily.

Conventionally, inorganic products that are manufactured of cement, gypsum, or clay or that are manufactured of a

compound in which an organic polymer or inorganic compound is combined with an inorganic composition already exist, but items manufactured of cement have the disadvantage that they are subject to low fracture toughness, low flex strength, occurrence of efflorescence, occurrence of constriction cracking, slow strength manifestation, etc. In addition, items manufactured of gypsum have the disadvantage that they have a low mechanical strength and are poor in water resistance and items manufactured of clay have the disadvantage that they must be fired at high temperatures to produce a high mechanical strength and that production yields are low as a result of deformation and cracking in the drying and firing stages. Moreover, inorganic products in which a water-dispersing organic polymer has been compounded with a cement - gypsum composition have been developed frequently in the prior art, but almost all of these have been poor in mechanical strength and water resistance, and the manufacture of those that have attained a fairly high strength (a flex strength of 200 kg/[illegible] or greater) and water resistance has required [illegible] stages such as autoclave treatment, pressing, or coating by UV or [illegible]B treatment as well as large facilities and equipment. This has presented problems with manufacturability or economy.

Thus, the easy manufacture of products that hardened items that have high strength and water resistance, and especially, products that are thin and have large dimensions has been extremely difficult.

The inventor has devised the present invention has solved the problems of conventional inorganic products described above by, in order to make the air bubble ratio and air bubble diameter in the hardened material as small as possible, (1) maintaining the fluidity of the slurry and holding the volume of mixed water [illegible] to a volume extremely close to the [illegible] water content or [illegible] water content by using an acryl water-dispersing organic polymer that has moisture-reducing and water-dispersing effects (2) generating ettringite fibers to further reduce water and filling the spaces in the hardened material with these crystals, and (3) by using a mixture of hydraulic cement and hydraulic gypsum to realize their mutually complementary effects. And has found that it demonstrates superior water resistance and strength.

That is to say, the inventor has provided the present invention that is a hydraulic inorganic composition that is characterized in that it comprises a) 10 - 90 parts by weight of a hydraulic cement, b) 10 - 90 parts by weight of a hydraulic

gypsum, c) 17 - 25 parts by weight of water (including the moisture contained in the acryl water-dispersing organic polymer of d)), d) 2 - 16 parts by weight of an acrylic, water-dispersing organic polymer that has a moisture-reducing effect (solid portion converted), and e) 0.5 - 2.0 parts by weight of a moisture-reducing agent.

The hydraulic cement according to the present invention may be a Portland cement, aluminate cement, white cement, blast furnace slag cement, silica fume cement, and so forth, commonly used in engineering construction, and may be used singly or in some combination thereof. The hydraulic gypsum may be a calcined gypsum (alpha form or beta form) or anhydrous gypsum and may be used singly or in some combination thereof. The ratio by weight of hydraulic cement to hydraulic gypsum is in the range 10:90-90:10, but when the hydraulic cement is less than 10 parts by weight or when the hydraulic gypsum is less than 10 parts by weight, the properties that are the objective of the present invention cannot be obtained, and especially, cracking, etc. has been observed when the hydraulic cement is greater than 90 parts by weight.

The water-dispersing organic polymer according to the present invention refers to a polymer in which minute particles

are dispersed homogeneously in water and which forms a socalled [illegible] latex or emulsion, and may be, broadly classified, a vinyl acetate, an acryl, a chlorine-containing vinyl polymer, a compound rubber, etc. However, when mixed with a hydraulic inorganic material, an acryl is preferred because it does not reduce the fluidity of the mixture and it produces a water-reducing effect, yet it still generates high strength, water resistance, etc., in the hardened material. In other words, copolymers of an acrylate ester and a methacrylate ester are indicated as the acryl water-dispersing organic polymer, but this includes copolymers that contain approximately one half or more of an acrylic monomer. Of these, a material that generates a water-reducing effect and maintains the fluidity of the slurry, that has a high film strength, and that is superior in water resistance, alkali resistance, [illegible] resistance, and polish is preferred, specifically methyl methacrylate-2ethylhexylacrylate, styrene-butyl acrylate, etc. Here, to obtain the required water resistance and high strength, the amount of [illegible] water is reduced as much as possible to approach the theoretical moisture content, but this can be accomplished by mixing moisture-reducing agents with these acryl waterdispersing organic polymers that possess moisture reducing

properties. However, little improvement in water resistance and strength, etc., is observed when the amount of acryl waterdispersing organic polymer used is 2 parts by weight or less. In addition, volumes of 16 parts by weight or more are disadvantageous from the viewpoint of cost, and since no improvement in strength is observed, the volume of acryl water-dispersing organic polymer with moisture-reducing effect that is used is in the range 2 - 16 parts by weight, preferably 4 - 12 parts by weight (both converted to solids), with respect to 100 parts by weight of a mixture of hydraulic cement and hydraulic gypsum. In this way, since the dispersibility of the mixture of hydraulic cement and hydraulic gypsum is improved and the moisture-reducing effect and strength appearance in the hardened material are further improved, a moisture-reducing agent for general cement use is used. Specific examples are sodium lignin sulfonate, sodium salts of melamine sulfonate formaldehyde condensate, sodium salts of β -naphthalene sulfonate formaldehyde condensate, sodium salts of creosol sulfonate formaldehyde condensate, etc., but sodium salts of melamine sulfonate formaldehyde condensate are most preferred.

The amount to be added is in the range 0.5 - 2.0 parts by weight, preferable 0.5 - 1.0 parts by weight, with respect to 100 parts by weight of the mixture of hydraulic cement and hydraulic gypsum.

An amount of mixed water that is the theoretical volume of water or is extremely close to the theoretical volume of water is sufficient and is in the range 17 - 25 parts by weight, preferably 17 - 20 parts by weight, with respect to 100 parts by weight of the mixture of hydraulic cement and hydraulic gypsum, but this includes the water content of the acryl waterdispersing organic polymer, and ac cording to the amount of acryl water-dispersing organic polymer used, the water content of this may be sufficient and the addition of water is unneeded, and even when needed, the amount of water added is up to a maximum of 15 parts by weight. Note that, even with 17 - 25 parts by weight of water, fluidity of the slurry is sufficiently maintained, foaming and defoaming is easy, poured shapes can be formed easily. The reason is that, with a water volume of less than 17 parts by weight, the amount of water required for hydrophilia with the hydraulic materials is insufficient, and with a volume of more than 25 parts by weight, the amount of water is excessive and sufficient physical characteristics cannot be obtained.

In the present invention, strengthening agents, fillers, etc.,

can be added to further improve the physical characteristics of the hardened material. Strengthening agents used may be inorganic materials such as glass fibers, slag fibers, rock wool, asbestos, etc., organic materials such as polypropylene, vinyl polychloride, polyester, polyamide, etc., fibrous strengtheners from woody fibers such as pulp, used paper, sawdust, flax, cotton, etc., or fine particle diameter powder strengtheners such as carbon black, aluminum hydroxide, calcium carbonate, magnesium carbonate, white carbon, titanium dioxide, etc. The volume of these strengtheners is in the range 0.5 - 10 parts by weight with respect to 100 parts by weight of the mixture of hydraulic cement and hydraulic gypsum. Additionally, fillers may be talc, mica, barite, [illegible] powder, etc. Further, suitable amounts of publicly known defoaming agents, hardening accelerating agents, hardening slowing agents, water-repelling agents, water-resisting agents, coloring agents, etc., may be added as needed. Moreover, the surfaces of hardened items formed from the water-dispersing inorganic composition according to the present invention may be treated with hard coating materials such as silicon or ceramic coating materials to form a membrane film that will further improve dirt resistance, abrasion resistance, damage resistance, chemical resistance, contaminant resistance, polish, water resistance, etc.

When manufacturing a hardened item from the water-dispersing inorganic composition according to the present invention, the fluidity of the slurry is very good, despite the low volume of water content, and foaming and defoaming can be conducted easily. As a result, items can be formed by pouring after excitation defoaming methods that add a defoaming agent and use a table vibrator or vacuum stirring foaming methods have been used. This slurry is self-leveling, so that the formation of flat items is especially easy. Hardened items removed from molds can be heated at 60 - 100°C for four hours or more following natural [illegible] in a humid environment. When an acryl water-dispersing organic polymer that cannot be formed at normal temperatures is used, heating to the range of minimum forming temperature to 100°C is needed

The water-dispersing inorganic composition according to the present invention does not require large scale facilities and equipment and hardened items can be obtained very easily at low cost and can be made into thin shapes with especially large dimensions. Moreover, these hardened items have high strength, are excellent in water resistance, incombustibility, dirt resistance, vibration absorption, etc. Moreover, it is characterized in that yields are high due to almost no deformation or cracking during

manufacture and shape reproducibility is extremely good because swelling and shrinkage is extremely small. In addition, it has the effect that hardened items with extremely high polish can be obtained when molds with mirror surfaces are used.

Accordingly, the water-dispersing inorganic composition according to the present invention can be used widely as a engineering material such as tiles, blocks, paving stones, [illegible], interior and exterior [illegible] wall materials, flooring materials, ceiling materials, platform materials, interior materials, novelties, sound materials, vibration-absorbing materials ([illegible] materials), etc.

Next, the present invention will be further explained using embodiments. Note that for flex strength tests, specimens were formed with the dimensions $40 \times 160 \times 8$ [illegible], and the test equipment used was a Shimazu Engineering Co., Ltd. Model *IS*500 autograph. Tests were conducted in accordance with JIS standards.

Embodiment 1

One part by weight (hereafter simply called "part") of a powdered water-reducing agent (a sodium salt of melamine sulfonate formaldehyde) was dissolved beforehand in 5 parts of water. To this, 20 parts of an ester acrylate copolymer emulsion (converted solids, 8 parts) were added to form an aqueous paste.

To this was added a mixture of 20 parts of α form calcined gypsum, 80 parts of Portland cement, and 3 parts of glass fibers (chopped strands). The resulting solution was agitated and stirred at 550 rpm for 5 minutes (during this period, a suitable volume of a silicon defoaming agent was dripped into the solution) to obtain a slurry with excellent fluidity. This slurry was defoamed on a table vibrator for 5 minutes and was then poured into a plastic mold with a mirror surface and was hardened. Following hardening and removal from the mold, the material was [illegible] at normal temperature, and was then heated for 4 hours at 70 - 75°C, and further, for 30 minutes at 90 - 95°C. This hardened item had a high polish, its flex strength was 306.5 kg/[illegible], and its absorbed water ratio (after submersion for 24 hours) was 0.51%. Embodiment 2

A slurry produced in the same manner as Embodiment 1 was poured into a glass [illegible] with the dimensions 440 x 500 [illegible] to a depth of 5 [illegible] to obtain a hardened item. When the item was [illegible] and heated under the same conditions as Embodiment 1, there was almost no swelling or shrinkage and no deformation or cracking, so that a high strength, high polish product with large dimensions was obtained.

Embodiment 3

0.5 parts of a powdered water-reducing agent (a sodium salt of melamine sulfonate formaldehyde) was dissolved beforehand in 5 parts of water. To this, 20 parts of an ester acrylate copolymer emulsion (converted solids, 8 parts) were added to form an aqueous paste. To this was added a mixture of 60 parts of α form calcined gypsum, 40 parts of aluminate cement, 5 parts of carbon black, and 3 parts of glass fibers (chopped strands). The resulting solution was agitated and stirred at 550 rpm for 5 minutes (during this period, a suitable volume of a silicon defoaming agent was dripped into the solution) to obtain a slurry with excellent fluidity. After pouring, defoaming, [illegible], and heated in the same manner as Embodiment 1, the material flex strength was 268.2 kg/[illegible], and its absorbed water ratio (after submersion for 24 hours) was 0.55%.

Patent Applicant: Inoue, Hiroyuki

9日本国特許庁(JP)

· 10 特許出 關 公 開

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審査請求 未請求 発明の数 1 (全4頁)

69発明の名称 水硬性無機質組成物

> **1049** 翻 昭59-28234

田田 顧 昭59(1984)2月16日

Ż 大田市大田町大田への263 之 大田市大田町大田への263 砂田 随

1. 発明の名数

- **b**) 10~90登景部
- 17~25重量部(但し、4)のア 含 to)
- 減水効果を有するアクリル系水分散性有 2~16 波量部(但し、图形分 及算) および
- 0.5~2.0重量部 から或ることを特徴とする、水硬性無偏質組成

3. 発明の静範な説明

本発明は、皮形、亀裂の発生がなく耐水性およ び高強度を発現し、大寸法で都形の硬化体をも簡 品に成形し得る水硬性無根質組成的に関する。

從前より無倒質製品としてはセメント、石膏、 粘土製のもの、あるいは無機組成物に有機型合体 または経機化合物を配合したもの等の既存製品が あるが、セメント製のものは低破壊じん性、低曲 げ強度、エフロレッセンスの発生、収縮角裂の発 、頭皮発現が遅い等の眼点があり、また石膏盤 ものは概線的強度が小さく耐水性に劣り、粘土 のものは領域的強度を大きくするためには高温 焼成が必要であり、乾燥、焼成工程における密形、 亀裂等の発生による歩智りの低さ等の能点がある。 そしてまた、セメントー石膏組成物に各種の水分 飲住有機食合体を配合した無機質製品は従前より 多数関発されているが、その殆んどは機械的強度、 水性等において劣っており、かなりの強度(血 度2.00 4 / 山以上) および耐水性を発現す に到った成形物は、その製造においてオ プ処理、プレス成形あるいはロV、エB処期 によるコーティング等の奴隷な工程および大きな 留顔器を受し、その生産性あるいは経済性にお

従って、高強度および耐水性を有する硬化体、なかんずく大寸法で養形の級品を簡易に製造するととは細めて困難であった。

すなわち本類明は、4)水硬性セメント10~ 90重量部、b)水硬性石膏10~90重量配、 。)水17~25重量部(但し、4)のアクリル 系水分数性有機重合体中の水分量も含む)、4) 減水効果を有するアクリル系水分数性有機重合体 2~16重量部(但し、配形分換算)および。) 減水剤 0.5~2.0重量部から成ることを特徴とす る水硬性組織質組成物を提供することにある。

本発明において水分数性有機型合体とは、その 類観粒子が水の中に均一に分数して、所謂ラテラ

゚゙ゥスまたはエマルジョンと呼ばれる形態になって ·Vaiるものを意味し、大別すると酢酸ピニル系、7 クリル系、塩素含有ビニルポリマー系、合成コム ・系布があるが、水硬性低機質材料と混合した時、 能合物の流動性を低下させず、減水効果を生じ、 しかも硬化体が高強度、耐水色等を発現するのは アタリル系のものが最良である。すなわち、アタ リル系水分数性有機重合体とは、アクリル酸エス ナルとメタクリル酸エスナルとの共型合体を指す が、程度半量以上のアクリルモノマーが含有され る共駆合体も包含される。中でも、波水効果を発 現するとともにスクリーの途動性を保持し、フィ ルム強度が大きく、耐水性、耐アルカリ性、耐快 性、光沢性に使れた特性を有するものが好ましく、 具体的にはメチルメタクリレートー 2-エチルヘキ シルアクリレート、スチレンープチルアクリレー 🖔 ト等が挙げられる。そとで所聞の耐水性および高 敦度尊を得るには、范水量を可能な限り減少させ 理論水分量に近ずけることであるが、彼水剤およ び旅水効果を有するとれらのアタタル系水分散性

有機重合体を混合することにより可能となる。し かし、アクリル系水分散性有機重合体の使用量が 2 重量部以下であると耐水性および強度等の向上 はわまり超められず、また16型量部以上ではコ スト面において不利になり、且つ強度の一層の肉 上は悶められないので、波水効果を有するアッリ ル系水分数性有機重合体の使用量は水裂性セメン トと水硬性石膏の混合物100重量部に対して2 ~16重量部、好ましくは4~12重量部(但し、 いずれも固形分換算)の範囲である。そして、水 **段性セメントおよび水硬性石膏の分散性を良くし** 低いては彼水効果および硬化体の強度発現を一層 大きくするために、遊常のセメント用波水剤が使 用される。具体的にはリグニンスルホン酸ナトリ ウム、メラミンスルホン酸ホルムアルデヒド組合 **効ナトリウム塩、βーナフォリンスルホン豊ホル** ムアルデヒド箱合物ナトリウム塩、クレゾールス ルホン酸ホルムアルザヒド自合物ナトリウム塩等 が挙げられるが、メラミンスルホン酸ホルムアル デヒド輪合物ナトリウム塩が最も好ましく、その

添加量は水硬性セメントと水硬性石膏の混合物 100重量部に対して0.5~2.0重量部、好せし くは0.5~1.0重量部である。

本発明において、優化体の物性を更に向上させ、 るために公知の補強材、完複材等を配合すること ができる。植強材としては、ガラス繊維、スラグ 趣 雄 、ロックサール 、石 錦 等 の 無 機 総 辞 や ポ り ブ ロビレン、ポリ塩化ビニル、ポリエステル、ポリ アミド等の有機機器、あるいはパルブ、放紙、木 別、席、結集の木質系統組から成る機能質情強材、 ·さらにカーメンプラック、水酸化アルミニウム、 **単独カルシウム、炭酸マグネシウム、ホワイトカ** ニメン、二酸化チタン等の微粒子径粉束の無強材 が使用できる。とれら精強材の配合量は、水硬性 セメントと水硬性石膏の総合物100重量部に対 して 0.5~10 重量部である。また、光模材とし ては、タルタ、マイカ、パーライト、陶石粉質が 使用される。そしてまた、公知の消泡剤、硬化促 追刺、硬化温袋剤、はっ水剤、耐水化剤、着色剤 等を必要に応じて適宜添加することができる。更 に、本発明に係る水硬性無機質組成物から得られ た硬化体の表面にシリコーン等のパードコート材 あるいはセラミックコーテイング材料を処理する ととにより被膜を形成し、耐保性、耐麻純性、耐 练每性、闭塞品性、耐污染性、光沢性、耐水性等

を一層向上させることもできる。

本現明に係る水炭性無機質組成物は、大規模を 機関を受することなく緩めて簡易かつ安価に設 化体を、をかんずく大寸法で薄形の硬化体をも符 ることができ、しかもその硬化体は高強度であり 耐水性、不識性、射像性、吸線性等に優れ、また その製造において変形、色製の発生が殆んどなく 終少割りで、態度収弱が極めて少なく型再現性が 非常に良好であるという物散を有する。そしてま た、観団を有する型を使用した場合には、極めて 高光沢の硬化体を得る効果をも有する。

従って、本発明に係る水硬性無機質組成物は、 タイル、プロック、数石、重視材、内外接触材、 中材、天井材、配物台材、インテリア材、ノベル ティ、音響材、振動吸収材(細細材)等の工業材料として広仇に利用できるものである。

次に実施例により本発明をさらに詳細に説明する。尚、曲げ強度試験は、試験片として40×160×8=06のを作成し、試験設置は(練) 品本製作所製のオートグラフェ8500窓を使用しょ18に則り実施した。

安全 倒 1

水 5 盆量部 (以下、単に部と称す。) に 形末状の 級水剤 (メラミンスルキン腹 ホルムアルデヒ ド 総合物ナトリウム塩) | 部を予め辞解し、 これにアクリル酸エステル系共成合体エマルジョン 2 0

特高昭60-171260 (4)

实施例 2

実施例1と同様にして副製したスタリーを440×500〜の大寸後のガラス製型に流込み厚さ5〜の硬化体を存在。この硬化体を突施例1と全く同様に透空養生養、加熱したところ影膜収縮が殆んどなく、変形、亀裂を全く生じない大寸法で容形の高效症、高光沢製品を得た。

实施贸3

本も都に粉束状の設ま剤(メラミンスルムン酸をホルムでルデヒド前合物ナトリウム塩ンの、5部を合作し、これにアクリル酸工算を発展している。 2 年間 の 1 と 2 年間 の 2 年間 の 3 年間 の 3 年間 の 3 年間 の 4 日間 の 4 日間 の 5 年間 の 6 日間 の 7 年間 の 7 年間

特許出賦人 井上博之